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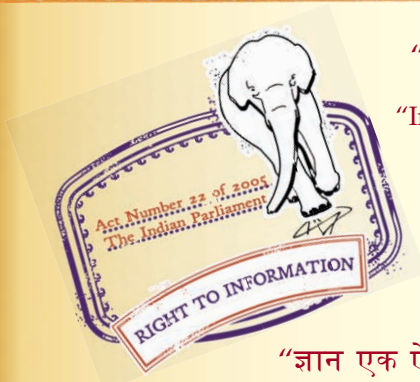
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IS 501 (1976): Oxalic Acid, Technical and Analytical Reagent [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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**IS : 501 - 1976**  
**( Reaffirmed 1990 )**

***Indian Standard***

**SPECIFICATION FOR  
OXALIC ACID, TECHNICAL AND  
ANALYTICAL REAGENT**

**( *Second Revision* )**

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**Second Reprint MARCH 1993**

**UDC 661.733.1**

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**BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002**

# Indian Standard

## SPECIFICATION FOR OXALIC ACID, TECHNICAL AND ANALYTICAL REAGENT ( Second Revision )

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*Indian Standard*  
SPECIFICATION FOR  
OXALIC ACID, TECHNICAL AND  
ANALYTICAL REAGENT  
( *Second Revision* )

0. FOREWORD

**0.1** This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 23 November 1976, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was originally published in 1953. It was subsequently revised in 1963 raising the requirement for minimum content of oxalic acid from 90.0 to 96.0 percent and incorporating requirements for various impurities in respect of technical grade, and making the requirements for impurities more rigid in respect of analytical reagent grade of the material. In the present revision the limit for minimum content of oxalic acid, technical has been further raised from 96.0 to 98.5 percent, and that for sulphated ash has been reduced from 0.5 to 0.15 percent. Limit for heavy metals has also been specified. For the analytical reagent grade the sulphate limit has been reduced.

**0.3** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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**1. SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for oxalic acid, technical and analytical reagent.

**2. REQUIREMENTS**

**2.1 Description** — The material shall be in the form of colourless crystals, free from efflorescence. When heated, it shall volatilize without charring.

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\*Rules for rounding off numerical values (revised).

**2.2 Solubility** — Dissolve 5.0 g of the material in 50 ml of warm water. A clear colourless solution shall be produced without sediment.

**2.3** The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

**TABLE 1 REQUIREMENTS FOR OXALIC ACID, TECHNICAL AND ANALYTICAL REAGENT**

Sl. No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST ( REF TO CL No. IN APPENDIX A )
		Technical Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Oxalic acid content [ ( COOH ) <sub>2</sub> . 2H <sub>2</sub> O ], percent by mass, <i>Min</i>	98.5	99.8	A-3
ii)	Sulphated ash, percent by mass, <i>Max</i>	0.15	0.01	A-4
iii)	Chlorides ( as Cl ), percent by mass, <i>Max</i>	0.1	0.001	A-5
iv)	Sulphates ( as SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.1	0.005	A-6
v)	Heavy metals ( as Pb ), percent by mass, <i>Max</i>	0.005	0.000 5	A-7
vi)	Nitrogen compounds ( as N ), percent by mass, <i>Max</i>	—	0.001	A-8
vii)	Iron ( as Fe ), percent by mass, <i>Max</i>	0.05	0.000 5	A-9
viii)	Calcium ( as Ca ), percent by mass, <i>Max</i>	—	0.002	A-10
ix)	Magnesium ( as Mg ), percent by mass, <i>Max</i>	0.1	0.003	A-10

### 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed preferably in glass bottles or as agreed to between the purchaser and the supplier.

#### 3.2 Marking

**3.2.1** The containers shall be marked with the following information:

- Name and grade of the material;
- Manufacturer's name and recognized trade-mark, if any;



- c) Net mass of the material; and
- d) Lot or batch number in code or otherwise.

**3.2.2** The containers for the analytical reagent grade shall also be labelled with full analytical data of the characteristics prescribed in Table 1.

**3.2.3** The containers may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1** Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

# APPENDIX A

( Clause 2.3 and Table 1 )

## ANALYSIS OF OXALIC ACID, TECHNICAL AND ANALYTICAL REAGENT

### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1960\* ) shall be used in tests.

**NOTE** — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

### A-2. PREPARED SOLUTION

**A-2.1** Dissolve 10.000 g of the material in 1 000 ml of water in a graduated flask. Use this *prepared solution* for test where so indicated.

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\*Specification for water, distilled quality ( revised ).

**A-3. DETERMINATION OF OXALIC ACID CONTENT****A-3.1 Reagents****A-3.1.1 Dilute Sulphuric Acid** — 1 : 1.**A-3.1.2 Standard Potassium Permanganate Solution** — 0.1 N, freshly standardized.

**A-3.2 Procedure** — Dissolve about 0.3 g of the material, accurately weighed, in 200 ml of freshly distilled water and add 10 ml of dilute sulphuric acid. Add, with stirring, 35 ml of standard potassium permanganate solution from a burette at a rate of 25 to 30 ml per minute. Heat to 55 to 60°C, stir and complete the titration by adding more of standard potassium permanganate solution till the colour persists for at least 30 seconds. Add the last 0.5 to 1 ml of standard potassium permanganate solution dropwise taking particular care to allow each drop to become decolourized before the next drop is added.

**A-3.3 Calculation**

Oxalic acid content [ (COOH)<sub>2</sub>.2H<sub>2</sub>O ], percent by mass =  $\frac{6.303 V N}{M}$

where

$V$  = volume in ml of standard potassium permanganate solution required for titration,

$N$  = normality of standard potassium permanganate solution, and

$M$  = mass in g of the material taken for the test.

**A-4. DETERMINATION OF SULPHATED ASH****A-4.1 Reagent****A-4.1.1 Concentrated Sulphuric Acid** — See IS : 266-1961\*.

**A-4.2 Procedure** — Weigh accurately about 10 g of the material in a weighed silica dish of about 20 ml capacity. Add about 0.5 ml of concentrated sulphuric acid and ignite gently. Cool in a desiccator and weigh. Repeat this process till successive weighings agree within 1 mg.

**A-4.3 Calculation**

Sulphated ash, percent by mass =  $\frac{100 M_1}{M_2}$

where

$M_1$  = mass in g of the residue, and

$M_2$  = mass in g of the material taken for the test.

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\*Specification for sulphuric acid (*revised*).

## A-5. TEST FOR CHLORIDES

### A-5.1 For Technical Grade

#### A-5.1.1 Apparatus

A-5.1.1.1 *Nessler cylinders* — 25 ml capacity.

#### A-5.1.2 Reagents

A-5.1.2.1 *Dilute nitric acid* — approximately 4 N.

A-5.1.2.2 *Silver nitrate solution* — approximately 4 percent (*m/v*).

A-5.1.2.3 *Standard chloride solution* — Dissolve 0.165 g of sodium chloride previously dried at  $105 \pm 2^\circ\text{C}$ , in water and dilute to 1 000 ml. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of this diluted solution contains 0.01 mg of chloride (as Cl).

A-5.1.3 *Procedure* — Take 5 ml of the *prepared solution* (A-2) in a Nessler cylinder, add 2 ml of dilute nitric acid and 1 ml of silver nitrate solution. In another Nessler cylinder prepare a control using 5 ml of standard chloride solution in place of the material. Dilute the contents of the two cylinders to the mark and shake them.

A-5.1.4 The material shall be taken to have not exceeded the limit prescribed in Table 1 if the opalescence produced with the material is not greater than that obtained in the control.

### A-5.2 For Analytical Reagent Grade

#### A-5.2.1 Reagents

A-5.2.1.1 *Dilute nitric acid* — same as in A-5.1.2.1.

A-5.2.1.2 *Silver nitrate solution* — same as in A-5.1.2.2.

A-5.2.2 *Procedure* — Weigh 2.0 g of the material and dissolve in 45 ml of warm water. Add 5 ml of dilute nitric acid and 0.1 ml of silver nitrate solution.

A-5.2.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if no opalescence is produced within 5 minutes.

## A-6. TEST FOR SULPHATES

### A-6.1 For Technical Grade

#### A-6.1.1 Apparatus

A-6.1.1.1 *Nessler cylinders* — 25 ml capacity.

#### A-6.1.2 Reagents

A-6.1.2.1 *Sodium carbonate*

**A-6.1.2.2 Hydrogen peroxide** — 30 percent.

**A-6.1.2.3 Dilute hydrochloric acid** — 4 N and 1 N.

**A-6.1.2.4 Barium chloride solution** — 10 percent.

**A-6.1.2.5 Standard sulphate solution** — Dissolve 1.240 g of ignited sodium sulphate in water and dilute to 1 000 ml. Dilute 10 ml of this solution again to 1 000 ml. One millilitre of this diluted solution contains 0.01 mg of sulphate ( as  $\text{SO}_4$  ).

**A-6.1.3 Procedure** — Take 5 ml of the *prepared solution* (A-2) in a platinum dish, add 10 ml of water and 0.5 g of sodium carbonate in small portions. Evaporate to dryness and ignite gently. Add to the residue 10 ml of water and 2 ml of hydrogen peroxide, and boil for 3 minutes. Add 3 ml of 4 N hydrochloric acid and evaporate to dryness on a steam-bath. To the residue add 5 ml of hot water, filter and wash the residue on the filter paper with water and make up the volume to 10 ml in a Nessler cylinder. Add 0.2 ml of 1 N hydrochloric acid and 2 ml of barium chloride solution. Prepare a control in another Nessler cylinder using 5 ml of standard sulphate solution in place of the *prepared solution* and the same quantities of other reagents. Dilute the contents of both the Nessler cylinders to the mark and compare the turbidity produced in the two cylinders.

**A-6.1.4** The material shall be taken to have not exceeded the limit prescribed in Table 1 if the turbidity produced with the material is not greater than that obtained in the control.

## **A-6.2 For Analytical Reagent Grade**

### **A-6.2.1 Reagents**

**A-6.2.1.1 Sodium carbonate**

**A-6.2.1.2 Hydrogen peroxide** — same as in A-6.1.2.2.

**A-6.2.1.3 Dilute hydrochloric acid** — approximately 4 N.

**A-6.2.1.4 Barium chloride solution** — same as in A-6.1.2.4.

**A-6.2.2 Procedure** — Dissolve 1.000 g of the material in 20 ml of water in a platinum dish. Add, in small portions, 1 g of sodium carbonate, evaporate to dryness and ignite gently. Add to the residue 15 ml of hot water and 5 ml of hydrogen peroxide, boil and cool. Add 6 ml of dilute hydrochloric acid and filter. To the filtrate add 30 ml of water and 2 ml of barium chloride solution and allow it to stand for 1 hour.

**A-6.2.3** The limit prescribed in Table 1 shall be taken as not having been exceeded if no turbidity or precipitate is produced.

## **A-7. TEST FOR HEAVY METALS**

### **A-7.1 Apparatus**

**A-7.1.1 Nessler Cylinders** — 50 ml capacity.

## A-7.2 Reagents

**A-7.2.1 Ammonium Hydroxide** — approximately 6 N.

**A-7.2.2 Concentrated Nitric Acid** — See IS : 264-1976\*.

**A-7.2.3 Sodium Sulphide Solution** — approximately 10 percent.

**A-7.2.4 Standard Lead Solution** — Dissolve 1.600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute it again with water to 1 000 ml. One millilitre of this diluted solution contains 0.01 mg of lead ( as Pb ). This diluted solution shall be prepared fresh.

**A-7.3 Procedure** — Weigh 2.000 g of the material in the case of analytical reagent grade and 0.2 g in the case of the technical grade and transfer to a Nessler cylinder. Dissolve it in 30 ml of water and neutralize with ammonium hydroxide. Acidify slightly with concentrated nitric acid. Add two drops of sodium sulphide solution and dilute to the mark. Prepare a control in another Nessler cylinder using 1 ml of standard lead solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour of the solutions 10 minutes after the addition of sodium sulphide solution.

**A-7.4** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that produced in the control.

## A-8. TEST FOR NITROGEN COMPOUNDS

### A-8.1 Apparatus

**A-8.1.1 Nessler Cylinders** — 50 ml capacity.

### A-8.2 Reagents

**A-8.2.1 Dilute Sodium Hydroxide Solution** — approximately 10 percent.

**A-8.2.2 Aluminium Wire**

**A-8.2.3 Dilute Hydrochloric Acid** — approximately 5 N.

**A-8.2.4 Nessler Reagent** — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly, with stirring, saturated mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide. When it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep the solution in a bottle closed with a well-fitting rubber stopper.

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\*Specification for nitric acid ( second revision ).

**A-8.2.5 Standard Ammonium Chloride Solution** — Dissolve 0.382 g of ammonium chloride in ammonia-free water and make up the volume to 1 000 ml. From this, pipette out 10 ml of the solution and dilute again with ammonia-free water to 100 ml. One millilitre of this diluted solution contains 0.01 mg of nitrogen ( as N ).

**A-8.3 Procedure** — Dissolve 2.00 g of the material in 20 ml of water and while cooling it in ice, add 4 ml of dilute sodium hydroxide solution. Add 0.5 g of aluminium wire in small pieces, and allow to stand for 2 hours protected from loss of or access to ammonia. Distil off about 40 ml of this solution into a Nessler cylinder containing 5 ml of water acidified with one drop of dilute hydrochloric acid. Add to the distillate 2 ml of dilute sodium hydroxide solution and 2 ml of Nessler reagent. Make up the volume to 50 ml. Prepare a control in another Nessler cylinder using 2 ml of standard ammonium chloride solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour developed in the two cylinders.

**A-8.4** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that produced in the control.

## **A-9. TEST FOR IRON**

### **A-9.1 Apparatus**

**A-9.1.1 Nessler Cylinders** — 50 ml capacity.

### **A-9.2 Reagents**

**A-9.2.1 Concentrated Hydrochloric Acid** — See IS : 265-1962\*.

**A-9.2.2 Dilute Sulphuric Acid Solution** — 10 percent ( v/v ).

**A-9.2.3 Ammonium Persulphate**

**A-9.2.4 Butanolic Potassium Thiocyanate Solution** — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

**A-9.2.5 Standard Iron Solution** — Weigh 0.702 g of ferrous ammonium sulphate [  $\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$  ] and dissolve in 10 ml of dilute sulphuric acid. Add 1 g of ammonium persulphate and heat to boiling. Cool and dilute with water to make up the volume to 1 000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre of this diluted solution is equivalent to 0.01 mg of iron ( as Fe ).

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\*Specification for hydrochloric acid ( revised ).

### A-9.3 Procedure

**A-9.3.1 For Technical Grade** — Transfer 5 ml of the *prepared solution* (A-2) to a Nessler cylinder. Add 1 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 seconds and allow the layers to separate.

**A-9.3.2 For Analytical Reagent Grade** — Dissolve 5.00 g of the material in 20 ml of water. Transfer the solution to a Nessler cylinder and carry out the test as prescribed in A-9.3.1.

**A-9.3.3** Prepare a control using 2.5 ml of standard iron solution and the same quantities of other reagents in the same total volume of the reaction mixture.

**A-9.4** The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the butanolic layer in the test with the material is not greater than that produced in the control.

## A-10. DETERMINATION OF CALCIUM AND MAGNESIUM

### A-10.1 Reagents

**A-10.1.1 Anhydrous Sodium Sulphate**

**A-10.1.2 Concentrated Sulphuric Acid** — See IS : 266-1961\*.

**A-10.1.3 Concentrated Hydrochloric Acid** — See IS : 265-1962†.

**A-10.1.4 Dilute Hydrochloric Acid** — approximately 4 N.

**A-10.1.5 Standard Calcium Solution** — Weigh 1.000 g of calcium carbonate dried at  $120 \pm 5^\circ\text{C}$  and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 000 ml in a graduated flask. One millilitre of the dilute solution is equivalent to 0.4 mg of calcium (as Ca).

**A-10.1.6 Standard Disodium Ethylenediamine Tetra-acetate (EDTA) Solution** — Weigh 3.72 g of disodium ethylenediamine tetra-acetate dihydrate in water and dilute in a graduated flask to 1 000 ml. The solution shall be standardized before use by the method given in A-10.2.2.

**A-10.1.7 Standard Sodium Hydroxide Solution** — 5 N.

**A-10.1.8 Murexide Indicator Solution (for Calcium Only)** — Grind 0.2 g of murexide with 10 g of sodium chloride until the mixture is homogeneous.

**A-10.1.9 Buffer Solution** — Dissolve 67.5 g of ammonium chloride in a mixture of 520 ml of ammonium hydroxide (relative density 0.92) and

\*Specification for sulphuric acid (*revised*).

†Specification for hydrochloric acid (*revised*).

250 ml of water. Dissolve a mixture of 0.931 g of disodium ethylenediamine tetra-acetate dihydrate and 0.616 g of magnesium sulphate heptahydrate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in 50 ml of water. Combine the solutions and make up to 1 litre. Five millilitres of this buffer solution added to 50 ml of distilled water should not consume more than one drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

**A-10.1.10 Eriochrome Black T Indicator Solution (for Calcium and Magnesium)** — Dissolve 0.1 g of the dye in 20 ml of methanol. This solution shall be prepared fresh every week.

## A-10.2 Procedure

**A-10.2.1** Mix 25.0 g of the material with 0.2 g of anhydrous sodium sulphate, add 5 drops of concentrated sulphuric acid and heat gently until the material is volatilized. Then ignite until no more fumes are produced and any carbon formed is oxidized. Heat the residue with 1 ml of concentrated hydrochloric acid and evaporate to dryness on a steam-bath. Dissolve the residue by warming with a mixture of 2 ml of dilute hydrochloric acid and 5 ml of water. Dilute in a volumetric flask to 100 ml. Use this solution for determination of calcium and magnesium.

### A-10.2.2 Standardization of EDTA Solution

**A-10.2.2.1 With murexide indicator** — Pipette out 50 ml of standard calcium solution (see Note) in a 250-ml conical flask and add to it 1 ml of standard sodium hydroxide solution to adjust the pH of the solution to about 12. Add 0.1 to 0.2 g of murexide indicator and titrate against standard EDTA solution until the colour changes from pink to violet blue.

NOTE — The aliquot for titration shall be neutral so that by adding standard sodium hydroxide solution or buffer solution (see A-10.2.2.2), the desired pH of the solution could be adjusted.

**A-10.2.2.2 With eriochrome black T indicator** — Pipette out 50 ml of standard calcium solution (see Note under A-10.2.2.1) in a 250-ml conical flask and add 5 ml of buffer solution to adjust the pH of the solution to approximately 10. Add 5 drops of eriochrome black T indicator and titrate against standard EDTA solution until the wine red colour changes to pure blue.

**A-10.2.3 For Calcium** — Pipette out 20 ml of the solution prepared as in A-10.2.1 in a 250-ml conical flask and dilute with 80 ml of water. Add 2 ml of standard sodium hydroxide solution followed by 0.1 to 0.2 g of murexide indicator. Titrate against standard EDTA solution until the colour changes from pink to violet blue. Note the burette reading  $V_1$ .

**A-10.2.4 For Calcium and Magnesium** — Pipette out 20 ml of the solution prepared as in A-10.2.1 in a 250-ml conical flask and dilute with 75 ml of water. Add 5 ml of buffer solution and 5 drops of eriochrome black T



indicator solution. Titrate against standard EDTA solution until the wine red colour changes to pure blue. Note the burette reading  $V_2$ .

### A-10.3 Calculation

a) Calcium ( as Ca ), percent by mass =  $20 V_1 A_1$

where

$V_1$  = volume in ml of standard EDTA solution used as in **A-10.2.3**, and

$A_1$  = mass in g of calcium equivalent to 1 ml of standard EDTA solution determined as in **A-10.2.2.1**.

b) Magnesium ( as Mg ), percent by mass =  $12.14 ( V_2 A_2 - V_1 A_1 )$

where

$V_2$  = volume in ml of standard EDTA solution used as in **A-10.2.4**,

$A_2$  = mass in g of calcium equivalent to 1 ml of standard EDTA solution determined as in **A-10.2.2.2**,

$V_1$  = volume in ml of standard EDTA solution used as in **A-10.2.3**, and

$A_1$  = mass in g of calcium equivalent to 1 ml of standard EDTA solution determined as in **A-10.2.2.1**.

## APPENDIX B

( Clause 4.1 )

### SAMPLING OF OXALIC ACID, TECHNICAL AND ANALYTICAL REAGENT

#### B-1. GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**B-1.1** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.2** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.3** The samples shall be placed in clean, dry and airtight glass or other suitable containers on which the material has no action.

**B-1.4** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.5** Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

## **B-2. SCALE OF SAMPLING**

**B-2.1 Lot**—In any consignment of one grade of the material, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one grade of the material is known to consist of different sizes of containers or different batches of manufacture, the containers belonging to the same size and batch shall be grouped together and each such group shall constitute a lot.

**B-2.2** For ascertaining the conformity of the material to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

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**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED  
FROM A LOT**

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 and above	10

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**B-2.3** The containers shall be selected at random and in order to ensure randomness of selection, procedures laid down in IS : 4905-1968\* may be followed.

## **B-3. PREPARATION OF TEST SAMPLES**

**B-3.1** From each of the containers selected according to **B-2.2** and **B-2.3**, a small representative portion of the material, about 60 g, shall be taken out with the help of a suitable sampling instrument.

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\*Methods for random sampling.

**B-3.2** Out of these portions, an equal quantity of the material shall be taken and thoroughly mixed to form a composite sample about 150 g in mass. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**B-3.3** The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples shall be marked for the purchaser, another for the supplier and the third to be used as referee sample.

**B-3.4** All the individual and composite samples shall be transferred to separate containers. These containers shall be sealed airtight and labelled with full identification particulars given in **B-1.5**.

**B-3.5** The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

## **B-4. NUMBER OF TESTS**

**B-4.1** Test for the determination of oxalic acid content shall be performed on the individual samples.

**B-4.2** Tests for all other characteristics given under **2** shall be performed on the composite sample.

## **B-5. CRITERIA FOR CONFORMITY**

**B-5.1 For Individual Samples** — From the test results for oxalic acid content, the mean  $\bar{X}$  and range  $R$  of test results shall be computed (range being defined as the difference between the maximum and the minimum values of test results). The lot shall be declared as conforming to the requirements of oxalic acid content if the value of the expression  $\bar{X} - 0.6 R$  is greater than or equal to the relevant value specified in Table 1.

**B-5.2 For Composite Sample** — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result of each of the characteristics shall satisfy the relevant requirement specified.

# BUREAU OF INDIAN STANDARDS

## Headquarters;

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanatha

( Common to all offices )

## Regional Offices:

	Telephones
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI-110002	[331 01 31 331 13 75]
*Eastern : 1/14 C.I.T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	36 24 99
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	[2 18 43 3 16 41]
Southern : C. I. T. Campus, MADRAS 600113	{41 24 42 41 25 19 41 29 16}
†Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093	6 32 92 95

## Branch Offices:

'Pushpak' Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	[2 63 48 2 63 49]
‡Peenya Industrial Area, 1st Stage, Bangalore Tumkur Road BANGALORE 560058	[38 49 55 38 49 56]
Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	6 67 16
Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002	5 36 27
53/5, Ward No. 29, R. G. Barua Road, 5th Byelane, GUWAHATI 781003	3 31 77
5-8-56C L. N. Gupta Marg ( Nampally Station Road ), HYDERABAD 500001	23 10 83
R14 Yudhister Marg, C Scheme, JAIPUR 302005	[6 34 71 6 98 32]
117/418 B Sarvodaya Nagar, KANPUR 208005	[21 68 76 21 82 92]
Patliputra Industrial Estate, PATNA 800013	6 23 05
T.C. No. 14/1421, University P.O., Palayam TRIVANDRUM 695035	[6 21 04 6 21 17]
<i>Inspection Office (With Sale Point) :</i>	
Pushpanjali, 1st Floor, 205-A West High Court Road, Shankar Nagar Square, NAGPUR 440010	2 51 71
Institution of Engineers ( India ) Building, 1332 Shivaji Nagar, PUNE 411005	5 24 35
*Sales Office in Calcutta is at 5 Chowringhee Approach, P.O. Princep Street, Calcutta 700072	27 68 06
†Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007	89 65 28
‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square Bangalore 560002	22 36 71

AMENDMENT NO. 1 JANUARY 1985

TO

IS:501-1976 SPECIFICATION FOR OXALIC ACID,  
TECHNICAL AND ANALYTICAL REAGENT

*(Second Revision)*

*(Page 3, clause 2.1, second sentence)* -  
Substitute the following for the existing  
sentence:

'When gradually heated, it shall volatilize  
without charring'

(PCDC 9)

**AMENDMENT NO. 2 JANUARY 1997**  
**TO**  
**IS 501 : 1976 SPECIFICATION FOR OXALIC ACID,**  
**TECHNICAL AND ANALYTICAL REAGENT**

*( Second Revision )*

[ Page 3, clause 2.1, second sentence ( see also Amendment No. 1 ) ] — Delete.

( Page 5, clause A-1.1 ) — Substitute 'IS 1070 : 1992\*' for 'IS : 1070 - 1960\*'.

( Page 5, foot-note with '\*\*' mark ) — Substitute 'Reagent grade water ( third revision )' for the existing matter.

( Page 6, clause A-4.1.1, and Page 11, clause A-10.1.2 ) — Substitute 'IS 266 : 1993\*' for 'IS : 266 - 1961\*'.

( Pages 6 and 11, foot-note with '\*\*' mark ) — Substitute '\*Sulphuric acid ( third revision )' for the existing matter.

( Page 10, clause A-9.2.1 ) — Substitute 'IS 265 : 1993\*' for 'IS : 265 - 1962\*'.

( Page 11, clause A-10.1.3 ) — Substitute 'IS 265 : 1993†' for 'IS 265 : 1962†'.

( Page 10, foot-note with '\*\*' mark, and Page 11, foot-note with '†' mark ) — Substitute 'Hydrochloric acid ( fourth revision )' for the existing matter.

( PCD 9 )